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When two bad data sets are better than one

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High-resolution synchrotron X-ray powder diffraction (SXPD) data alone are sometimes not enough to solve the structure of a complex polycrystalline material. Such was the case for the high-silica zeolites SSZ-61 and SSZ-87, where combining data from different sources, in particular XPD and electron microscopy, was vital to success. For SSZ-61, the SXPD data feature broad peaks and a resolution of ca. 1.2 Å. Although the pattern could be indexed, structure determination failed both with the charge flipping routine in SUPERFLIP [1] and with the zeolite-specific program FOCUS [2]. The unit cell parameters and HRTEM images indicated a relationship with ZSM-12 (MTW) and SSZ-59 (SFN), so several models derived from these two frameworks were built. Eventually, after considering Si-29 MAS NMR data and the size of the organic structure directing agent (SDA), a framework model that fits all the data emerged. To complete the structure, the SDA was included as a rigid-body, and its location and orientation optimized using simulated annealing. Subsequent Rietveld refinement confirmed the structure. In contrast to SSZ-61, the SXPD pattern for SSZ-87 was quite good, and it could be indexed with a C-centered cell. However, structure solution failed, probably because of the very high degree of reflection overlap (93%). Therefore, rotation electron diffraction (RED) data [3] were collected, but they proved to be of low resolution and poor quality. Only 2 of the 7 data sets could be indexed, and these had different unit cells. Neither fit the XPD pattern directly. The problem was traced to large errors in the RED cell parameters, and eventually one RED cell could be transformed to one similar to the SXPD cell. The RED data with this cell was only 15% complete up to a resolution of 1.22 Å. Even so, the structure could be solved using a recently developed version of FOCUS that works with ED data. The SDA was found as for SSZ-61, and the structure then confirmed by Rietveld refinement.

[1] *L. Palatinus, G. Chapuis, Journal of Applied Crystallography, 2007, 40, 786-790,* ***[2]*** *R. W. Grosse-Kunstleve, L. B. McCusker, C. Baerlocher, Journal of Applied Crystallography, 1997, 30, 985-995,* ***[3]*** *D. Zhang, P. Oleynikov, S. Hovmöller, et al., Zeitschrift für Kristallographie - Crystalline Materials, 2010, 225, 94–102; W. Wan, J. Sun, J. Su, et al., Journal of Applied Crystallography, 2013, 46, 1863-1873*

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